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### DEVELOPMENT OF ULTRA-THIN FILM PRESERVATIVE COMPOUNDS

27 May 1963

Prepared under Navy, Bureau of Naval Weapons

Contract NOw 63-0293-c

QUARTERLY REPORT NO. 2

Covering Period: 1 Feb. 1963 thru 31 Apr. 1963

FOSTER D. SNELL, INC.

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QUARTERLY REPORT NO. 2

1 February 1963 through 30 April 1963

FOSTER D. SNELL, INC. 29 W. 15th St., N. Y. 11, N. Y.

This report applies to work on:

Contract NOw 61-0855-c

#### ABSTRACT

Inhibitor-modified films of cellulose acetate butyrate and Acryloid B-72 afford generally good corrosion protection under cyclic condensation conditions, but have poor adhesion to metal surfaces.

Nonyl phenoxy acetic acid is an effective inhibitor in acrylic, cpoxy and cellulose acetate latyrate films, while Alox 2028 is effective in Piccopale 100 films.

Films tested in the controlled cyclic condensation humidity cabinet which gave promise of affording a high degree of protection from corrosive attack were applied to test panels and exposed in a louvered outdoor shed. Except for Ucar R-104 (a heat-cured organo-functional silicone), these films, after thirty days exposure, show good corrosion protection of steel.

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#### I. INTRODUCTION

Previous work on this and the preceding contract indicated that considerable protection is obtained with various corrosion inhibitors in very thin film applications. However, most of these materials do not form adequate, integral, non-tacky films as one of the desired objectives of this project. Some film-forming materials such as epoxy and acrylic resins give a measure of corrosion protection to metal surfaces though not as much as desired.

In order to improve the protection afforded by materials which form the desired films, corrosion inhibitors which proved effective were compounded with these materials. Initially, the film-forming materials selected for this study were an epoxy resin (Epon 1007), an acrylic resin (Acryloid B-72), a cellulose acetate butyrate polymer (EAB-381) and an inert hydrocarbon resin (Piccopale 100).

Quite effective were: ethylene glycol monostearate, Glycomul MA

(sorbitan esters of fatty acids), Armeen C (a mixture of primary, secondary and tertiary aliphatic amines), zinc linoresinate, Alox 2028 (a mixture of oxy acids and esters derived from oxidation of petroleum fractions containing a sulfonate inhibitor) and nonyl phenoxy acetic acid.

Ethylene glycol monostearate forms a powdery, non-film-forming substance on metal surfaces but nevertheless inhibits corrosive attack in the environment of the corrosion test chamber used in this work. Glycomul MA,

Armeen C and Alox 2028 deposit on metals as waxy films of varying clarity (good for Alox 2028, poor for Glycomul MA and Armeen C). Nonyl phenoxy acetic acid and zinc linoresinate deposit as tacky, transparent amber-colored films on steel.

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In order to evaluate the proposed formulations, the length of testing in the controlled cyclic condensation humidity cabinet was increased to 7 days. Exposure of test panels in an outdoor louvered shed was also begun at this time.

#### II. CORROSION INHIBITOR COMPATIBILITY

Ethylene glycol monostearate was grossly incompatible in all formulations with Epon 1007, Piccopale 100, Acryloid B-72 and Half-Second Butyrate - the film-formers selected for the current evaluation.

Alox 2028 was quite compatible with Piccopale 100, and was compatible in solution with the other polymers. However, Alox 2028 modified films of Acryloid B-72, Epon 1007, and Half-Second Butyrate were very hazy. Wettability of the Alox modified Piccopale resin solution appeared to be somewhat improved for the metal test surface.

Nonyl phenoxy acetic acid, on the other hand, was compatible with Epon 1007, Acryloid B-72 and Half-Second Butyrate and incompatible with Piccopale 100.

Half-Second Butyrate films could not tolerate zinc linoresinate modification, and Acryloid B-72 and Piccopale 100 films were striated slightly indicating partial incompatibility. The wettability of the Piccopale 100 for metal seemed adversely affected. Zinc linoresinate was compatible in Epon 1007 films.

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Limited compatibility was obtained with Armeen C and Piccopale 100 and Acryloid B-72. Half-Second Butyrate films could not accept Armeen C modification, while Epon 1007 films showed good receptivity for addition of Armeen C.

Compatibility of Glycomul MA with Epon 1007, Acryloid B-72 and Half-Second Butyrate was apparently without significant benefit. The adhesion of acrylic and cellulose acctate butyrate films was adversely affected and epoxy films clouded more than usual in the test chamber. Glycomul MA was not compatible in Piccopale 100 films.

#### III. CORROSION PREVENTION OF INHIBITED FILMS

Preparation of test specimens for corrosion testing in the controlled cyclic condensation humidity cabinet and louvered shed was accomplished as described in the first quarterly report on this project.

Operation of the humidity chamber for this period of testing was as described in the first quarterly report, but the length of test was increased from 2 days to 7 days.

#### A. PICCOPALE 100 COMPOSITIONS.

Piccopale 100 is a mixture of straight chain aliphatic hydrocarbon resins containing considerable cyclic, but no aromatic structures.

This pale, thermoplastic resin is derived from high temperature cracking of petroleum, and is produced from a mixture of unsaturated monomeric hydrocarbons of an average molecular weight of 90 composed chiefly of dienes and reactive olefins.

Unmodified, thin films of Piccopale 100 are of little value in protecting steel surfaces from rusting as shown in Table I of this report. Modifying the Piccopale 100 film composition by adding zinc linoresinate and Armeen C produce no beneficial results; in fact, an impairment of the wettability character of these films on metal surfaces occur. Inclusion of as little as 5 per cent Alox 2028, however, markedly increases the corrosion protection afforded by Piccopale 100 films. In two days of testing the amount of surface rust was reduced from 10 per cent of pitted surface area to about 2 per cent pitted surface area. Pits were small and scattered (less than 1/32 inch in diameter). Increasing the amount of Alox 2028 modification of Piccopale 100 films resulted in a slight improvement in protective ability of this composition. With 25 per cent modification, test panels after two days in the test chamber, showed only rare pitting (less than 5 small - 1/32 inch diameter - pits on any test surface), and after 7 days pitting approached that observed with films of 5 per cent modification after two days of testing. Wetting of steel by Piccopale 100 compositions containing Alox 2028 is also markedly improved and probably accounts for at least some of the improved corrosion protection of these films.

After about five days in the humidity cabinet, Piccopale 100 films with Alox 2028 modification tend to develop very small, uniform blisters. This condition may be due to some slight solvent entrapment in the films and, if so, can probably be corrected by formulating these compositions with higher boiling solvents.

One month of louvered shed exposure for specimens coated with Piccopale 100-Alox 2028 (75/25) films resulted thus far in very slight pitting (one to three pits on horizontally and angular mounted panels, none on vertically mounted panels).

#### B. ACRYLOID B-72 COMPOSITIONS

Unmodified Acryloid B-72 films give a modest degree of protection to steel surfaces against corrosion. Films of 0.4 mil thickness allow 2 to 4 per cent of the steel surface to be pitted after two days of humidity cabinet testing. Two per cent surface rusting, when it is of small pits - less than 1/32 inch diameter - indicates 4-8 pits observed on the test surface; four per cent indicates 12-16 small pits; up to 10 per cent pit surface rusting is roughly estimated in this fashion.

Formulation of Acryloid B-72 compositions with Armeen C result in films which allow 10 per cent test surface pitting (with 5 per cent Armeen C in the film) and about 5 per cent surface pitting with ten per cent film modification. These pits are few, but relatively large - up to 1/4 inch in diameter. Adhesion of these films is poor - the films are able to be stripped from the panels with little effort in large sheets.

Modification of Acryloid B-72 with 0. 4 per cent zinc linoresinate results in a striated film which, after two days of humidity cabinet
exposure, results in thin rust streaks up to one-half inch long that appear to
follow the film striations. Despite the partial incompatibility of zinc linoresinate and Acryloid B-72, adhesion of this film to the metal surface is quite
good - equivalent to that of the unmodified acrylic film. Panels coated with

films of this composition were exposed in the louvered shed in order to obtain some correlation data. After 30 days, however the rusting observed seems much less than anticipated - about the same degree of pitting is observed as with the Piccopale 100-Alox 2028 coated panels.

Glycomul MA added to Acryloid B-72 compositions results in a film which gives good protection against corrosion - only two or three small pits were observed on test panels after seven (7) days in the humidity chamber. However, the adhesion of Glycomul MA modified Acryloid B-72 to steel is extremely poor.

Acryloid B-72 films with nonyl phenoxy acetic acid protect metal to the same degree as Glycomul MA modified films; the effect on film adhesion is less severe although still considered to be adverse.

A composition (65) consisting of Acryloid B-72 compounded with Armeen C and zinc linoresinate yields a film which gives better protection than films from compositions containing only one of the additives.

However, adhesion of this film is poor, being about equivalent to the adhesion of films containing only Armeen C.

#### C. HALF-SECOND BUTYRATE (EAB-381) COMPOSITIONS.

Cellulose acetate butyrate films studied in this project are composed of 37 per cent combined butyryl groups, 13 per cent combined acetyl groups and 50 per cent combined cellulose residue which contains 2 per cent free hydroxy groups. These films (both the half-second viscosity grade and twenty-second grade) exhibit poor adhesion to the polished steel test surfaces and, unmodified, do not have any particular merit in thin films as metal protectants. While unmodified the twenty-second viscosity grade of cellulose acetate butyrate has poorer adhesion, it appeared to offer greater protection than did the half-second grade films. Rust creepage under the film seems particularly bad with half-second coatings. There is perhaps a slight degree of this creeping under twenty-second films as evidenced by growth of some of the pits to as large as 1/4 inch diameter.

Glycomul MA considerably improved the protection of Half-Second Butyrate films for steel, but still permitted edge rust to creep somewhat under the film. In addition, there was some clouding of these films on exposure to the conditions in the test chamber. Glycomul MA modification had no beneficial effect on the protection given by twenty-second viscosity grade films and aggravate the adhesion deficiencies of this film.

Compounding of Half-Second Butyrate with nonyl phenoxy acetic acid results in films which markedly reduce the degree of rusting although the adhesion of these films is still poor. The same, or slightly greater, degree of pitting is observed on test panels coated with Half-Second Butyrate-nonyl phenoxy acetic acid compositions as is observed on Acryloid B.72-nonyl phenoxy acetic acid composition coated panels. Panels coated with this Half-Second Butyrate composition exposed in the louvered shed show practically no signs of corrosive attack after one month - only two very small pits appearing on a horizontally mounted panel.

#### D. EPON 1007 COMPOSITIONS.

Epon 1007 is a solid high-molecular weight bisphenol Aepichlorohydrin-type epoxy resin of relatively low epoxide reactivity (epoxide
equivalent 1600-2000). Uncured films give moderate protection in humidity
cabinet testing. Adhesion of epoxy films to metal substrates is excellent,
but there is a tendency for very thin films to pinhole. It is necessary to
use a slow evaporation rate solvent system to overcome or minimize pinholing. Thin films of uncured Epon 1007 protect steel approximately as much
as do unmodified Acryloid B-72 films.

Although zinc lineresinate was quite compatible with Epon 1007, no advantage in corrosion protection accrued to films of Epon 1007 with zinc lineresinate. Protection of steel in the cyclic humidity chamber, in fact, decreased with these films. Clouding of films appeared greater than observed with unmodified Epon 1007 films. (Uncured Epon 1007 films cloud after 2-3 days exposure in the test chamber).

Epon compositions with Glycomul MA deposit films which give the same order or, perhaps, slightly better protection than unmodified Epon 1007 films. The films have the same tendency to cloud as do the unmodified films during accelerated testing.

Addition of Armeen C to Epon 1007 formulations improves the protective ability of these epoxy films. Five per cent of Armeen C in Epon 1007 films gives films which permit 5 to 8 very small pits on test panels after 7 days in the controlled cyclic condensation humidity cabinet. Increasing the amount of Armeen C modification to ten per cent reduced the number of pits observed to 2-3 per test panel. There was no evidence of film cloudiness as observed with other Epon 1007 composition films during this one week test period.

Ten per cent of nonyl phenoxy acetic acid in Epon 1007 films yields results similar to those obtained with Epon 1007 compositions containing ten per cent of Armeen C modification. Again, no film clouding was observable.

A composition (66) containing 92.2 parts of Epon 1007, 7.2 parts of Armeen C and 0.6 parts of zinc linoresinate left a film which gave excellent protection against corrosion (2 or 3 very small pits on several test panels), but clouded after a few days. In those instances where films clouded, it was necessary to remove the films in order to observe the condition of the test surfaces.

Unmodified Epon 1007 and Armeen C-Epon 1007 compositions were applied to panels exposed in the louvered, outdoor shed. As with most other samples under exposure, there are only 2 or 3 very small pits on a few of the panels after 30 days of exposure.

#### IV. PROPRIETARY SILICONE COMPOSITIONS

During the current report period, several proprietary silicone compositions were included in these studies. Two, Ucar R-101 and Ucar R-104, compositions of Union Carbide Company, require baking to obtain optimum properties. General Electric's SR-53 Silicone Resin does not require baking to be effective.

All Ucar R-101 and Ucar R-104 coated panels during this test period were baked at 115°C. for 30 minutes. This appears to have been a good schedule for Ucar R-101 coated panels, but may not have been sufficient for Ucar R-104 films. Ucar R-101 films show excellent adhesion to metal surfaces and give excellent protection during humidity cabinet exposures to test surfaces on which they are applied. The excellent protection is comparable to results obtained with some of the better Epon 1007, Acryloid B-72, Piccopale 100 compositions. However, there appear to be no deficiencies in the Ucar R-101 films such as have been noted in some of the other compounded films - (e.g., blistering - Piccopale 100, poor adhesion - Acryloid B-72). One month exposure of Ucar R-101 coated panels in the louvered shed has not resulted in any evidence of corrosion.

Panels coated with Ucar R-104 films did not perform as well as did Ucar R-101 coated panels in the 7 day cyclic humidity cabinet exposure. The protection was generally good, but there was some very slight underfilm rust-creep indicating a potential deficiency in adhesion. The Ucar R-104 coated panels failed rather suddenly after two weeks of exposure in the louvered shed. There was considerable pitting on the panel surfaces which was fairly uniformly spaced and may have been due to pinholing in the films, perhaps caused by windblown dust particles.

SR-53 Silicone Resin coated panels evidenced numerous (30-40 per panel) small pits at the end of one week in the humidity cabinet. Baking of SR-53 Silicone Resin films at 115°C. for 30 minutes resulted in panels with somewhat less pitting at the end of the last period (10-15 small pits per panel). SR-53 Silicone Resin coated panels exposed in the louvered, shed show no evidence of failure after 30 days.

#### V. LOUVERED SHED EXPOSURES

A louvered shed was constructed conforming to the measurements listed in Specification MIL-C-16173c. This was placed on the roof of 29 West 15th Street, New York City and provisions were made for mounting test panels in horizontal, vertical and slanted positions.

During this initial period of panel exposures, mean temperatures have averaged about 45°F. and the relative humidity was rather low for the most part. There were three days of precipitation during this exposure period.

Except for the apparently anomalous failure of Ucar R-104 coated panels, there have been no distinguishing results to date. Most of the panels show only a minimal amount of small pitting (no more than 5 pits on any one panel and on many panels there were no signs of rust). Uncoated polished panels are completely covered with a light rust film after one day.

Warmer temperatures and higher humidities will probably accelerate corrosive attack and film degradation on test panels.

#### VI. SUMMARY

During the current quarter, ultra-thin film preservative compounds were formulated with selected film-formers (Piccopale 100, Acryloid B-72, Half-Second Butyrate and Epon 1007 resins) in combination with previously tested corrosion inhibitors (Alox 2028, Armeen C, zinc linoresinate, Glycomul MA, and nonyl phenoxy acetic acid). Ethylene glycol monostearate was also included as an additive but could not be studied because of gross incompatibility with the above film-formers.

Alox 2028 was compatible only in Piccopale 100 compositions.

Despite a blistering problem due to solvent entrapment, the Alox 2028
Piccopale 100 blend proved to be an effective corrosion inhibitor system.

Zinc linoresinate, a very effective inhibitor of corrosion, was of little benefit in any of the compositions in which it found use. Glycomul MA, likewise an excellent inhibitor, was only partly beneficial to the resin compositions in which it was employed as an additive. In Acryloid B-72 compositions, there was improved corrosion resistance with Glycomul MA, but there was a severe deterioration of adhesion properties of these films compared with unmodified Acryloid B-72 films. There was very little effect on the performance of Epon 1007 compositions due to the inclusion of Glycomul MA. There was also mixed results in the modification of Half-Second Butyrate

compositions with Glycomul MA. Improved corresion resistance was accompanied by film clouding.

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Armeen C was most effective in Epon 1007 compositions, improving the corrosion resistance of these films and eliminating the clouding of films exposed in the cyclic humidity cabinet. Armeen C was of little use in Acryloid B-72 compositions, a possible slight increase in protection being more than offset by adverse effects on adhesion of these films.

Nonyl phenoxy acetic acid was the most effective inhibitor incorporated into the various resinous compositions. While incompatible with Piccopale 100, it provided as good or better improvement in corrosion resistance of Acryloid B-72, Epon 1007, and Half-Second Butyrate compositions as any other additive evaluated. The only drawback is the reduced adhesion observed with Acryloid B-72 films modified with this additive. This adverse adhesion effect, however, was no greater than with other additives employed in Acryloid B-72 compositions.

Three proprietary silicone compositions were also studied at this time. Of these, Ucar R-101, an organo functional silicone of Union Carbide, was very effective, while the other two were not as effective as some of the compositions mentioned earlier. The Ucar R-101 films were baked in order to obtain optimum properties. The need for heat treatment, plus high cost, may be a severe disadvantage for this material.

Outdoor shed storage of coated panels has begun, but exposures have not been sufficiently long to permit us to make any useful comparisons and evaluations.

#### VII. CONCLUSIONS

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The following compositions protect steel from atmospheric corrosive attack at least moderately well.

| Code | Composition               | Parts By Wt. |
|------|---------------------------|--------------|
| 39   | Piccopale 100             | 75           |
|      | Alox 2028                 | 25           |
| 54 . | Ucar R-101                |              |
| 49   | Epon 1007                 | 90           |
|      | Armeen C                  | 10           |
| 60   | Epon 1007                 | 90           |
|      | Nonyl Phenoxy Acetic Acid | 10           |
| 52   | Half-Second Butyrate      | <b>9</b> 0   |
|      | Nonyl Phenoxy Acetic Acid | 10           |
| 58   | Acryloid B72              | 90           |
|      | Nonyl Phenoxy Acetic Acid | 10           |

Nonyl phenoxy acetic acid is a very effective inhibitor in films in which it can be made compatible.

#### VIII. FUTURE WORK

Since the adhesion of Acryloid B-72 and Half-Second Butyrate compositions which have good corrosion properties is suspect, this property must be improved if they are to find use as ultra-thin film preservative compounds. An alkyd resin, FCD-555B (France, Campbell & Darling, Inc.) has been used to promote adhesion of Half-Second Butyrate films in clear metal lacquers and will be incorporated in further formulations involving Half-Second Butyrate compositions. Formulation modifications, such as resinblending will be used to improve the adhesion of Acryloid B-72 compositions.

Additional materials, including oxidation inhibitors as well as corrosion inhibitors, will be incorporated into test compositions. While other resins may be screened, primary effort will be directed to improving compositions based on those resins studied in this report.

Effects of handling panels and effect on lubricity of films will be included in further testing as part of the evaluation of candidate compositions.

Respectfully submitted,

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WM/BB:hn Oz. May 27, 1963

IX. RESULTS OF CORROSION TESTS

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|          |  | Thick           | Envi- | Test   | Condition of Panel                                       |                                     |
|----------|--|-----------------|-------|--------|--|-------------------------------------|
|          |  | ness            | ron   | length | 1  |                                     |
| Code     | Film   | (mils)          | ment  | (days) | rusted Type of Rust                                      | Remarks                             |
|          |  |                 |       |        |  |                                     |
| 21       | Piccopale 100  | <b>&lt;0.</b> 5 | ပ     | 2      | 10% Unevenly pitted (small pits)                         | •                                   |
| 21       | Piccopale 100  | <0.1            | ၁     | 7      | 75% Generally pitted(small pits)                         | Poer film                           |
| 34       | Acryloid B-72  | 0.4             | υ     | 2      | 2-4% Scattered (small) pitting                           | 1                                   |
| 19       | Cellulose acetate butyrate<br>EAB 381 (balf-second)  | <0.5            | υ     | 2      | 100% Uniformly heavily rusted with underfilm creepage    | Poor adhesion;<br>film peels freely |
| 19       | Cellulose acetate butyrate<br>EAB 381 (Half-second)  | 0.3             | υ     | 7      | 50% Generally heavily rusted with underfilm creepage     | Poor adhesion;<br>film peels freely |
| <u>5</u> | Cellulose acetate butyrate<br>EAB 381-20 (20-second) | 0.1+            | υ     | Ĺ      | 10% Scattered pitting (varying small to large (1/4"diam) | Poor adhesion;<br>film peels freely |
| 33       | Epon 1007  | <0.5            | υ     | 2      | 2-4% Scattered (small) pitting                           | 1<br>1                              |
| 33       | Epon 1067  | 0.2             | ၁     | 2      | 2-5% Scattered (small) pitting                           | 1                                   |
| . 33     | Epon 1007  | 0.2             | υ     | 7      | 5% Scattered (small) pitting                             | Film whitened;                      |
| 33       | Epon 1007  | 0.2             | တ     | 30     | None   | Exposure continuing                 |
| 11       | Alox 2028  | <0.5            | ນ     | 2      | 0%   | Waxy film                           |
| 25       | Zinc Linoresinate                                    | >0.5            | υ     | 2      | %0   | Tacky film                          |
| 25       | Zinc Linoresinate                                    | 0.1             | ၁     | 2      | 2% Scattered (small) pitting                             | Tacky film                          |
| 25       | Zinc Linoresinate                                    | ~0.5            | ၁     | 2      | 1% Rare (small) pitting                                  | Tacky film;<br>poor, uneven film    |
|          |  |                 |       |        |  |                                     |

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|          |   | Thick          | Envi-        | Test     | Condition of Panel                            |                            |
|----------|---|----------------|--------------|----------|---|----------------------------|
| Code     | Film  | mess<br>(mils) | ron-<br>ment | (days)   | 'ype of Rust                                  | Remarks                    |
| 23       | Armeen C (mixture of fatty amines)              | > 0.5          | υ            | . 2      | 0%  | Waxy film                  |
| 23       | Armeen C (mixture of fatty amines)              | 0.1            | υ            | 2        | 1% Rare (small) pitting                       | Waxy film                  |
| 8        | Nonyl phenoxy acetic acid (NPA)                 | <0.5           | IJ           | 2        | 0%0   | Tacky film                 |
| m        | Glycomul MA<br>(Sorbitan esters of fatty acids) | < 0.5          | ပ            | 2        | V 0%0   | Waxy film                  |
| ~        | Ethylene glycol monostcarate                    | <0.5           | ၁            | 2        | 0%  | Powdery; no film           |
| 37       | Piccopale 100 95. 0<br>Alox 2028 5. 0           | 0.2            | ၁            | 2        | 2% Scattered (small) pitting                  |                            |
| & E -24- | Piccopale 100 85.0<br>Alox 2028 15.0            | 0. 1           | ၁            | 2        | 2% Scattered (small) pitting                  |                            |
| 39       | Piccopale 100 75.0<br>Alox 2028 25.0            | 0.2            | Ŋ            | 7        | 1% Rarc (small) pitting                       |                            |
| 39       | Piccopale 100 75.0<br>Alox 2028 25.0            | 0.2            | ၁            | 7        | 2% Scattered (small) pitting                  | Film blistered<br>slightly |
| 39       | Piccopale 100 75.0<br>Alox 2028 25.0            | 0.2            | တ            | 30       | Scattered pitting (very small)                | Exposure<br>continuing     |
| 45       | Piccopale 100 99.1 Zinc Linoresinate 0.9        | <b>~0.</b> 1   | ပ            | <b>!</b> | Generally pitted 60% (varying small to large) | Poor film                  |
| 46       | Piccopale 100 Zinc Linoresinate 1.8             | <0,1           | υ            | 2        | Generally pitted 75% (varying small to large) | Poor film                  |
| 47       | Piccopale 100 Zinc Linoresinate 2.7             | <0.1           | ນ            | 7        | Generally pitted 75% (varying small to large) | Poor film                  |
|          |   |                |              |          |   |                            |

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|  | (muls)       |  | (days)   | rusted               | rusted Type of Rust  | Remarks   |              |
|--|--------------|--|--|----------------------|--|---|--------------|
| Piccopale 100 95. 0 Armeen C 5. 0                      | <b>40.</b> 1 | U  | 7  | 75%                  | Generally pitted<br>(varying, small to large)  | Poor film   |              |
| Alox 2085  | ~0.2         | U  | 2  | %                    |  | Oily  |              |
| Alox 2017  | ~0.2         | U  | 2  | %                    |  | Oily  | . =          |
| Piccopale 100 90, 0<br>Armeen C 10, 0                  | <b>C</b> 0.1 | U  | 2  | %06                  | Generally pitted<br>(varying, small to large)  | Poor film   |              |
| Piccopale 100 80. 0<br>Armeen C 20. 0                  | 0 < 0.1      | U  | 7  | <b>%06</b>           | Generally pitted<br>(varying, small to large,<br>mostly large)   | Poor film   |              |
| Acryloid B-72 99, 6<br>Zinc linoresinate 0.4           | 0.3          | υ  | 2  | 15%                  | Rust streaks (thin lines)  | Rust streaks<br>appear to follow<br>striations in film  | •            |
| Acryloid B-72 99. 6<br>Zinc linoresizate 0.4           |              | Ŋ  | 30   | ,                    | Rare pitting (small)<br>Horizontal and 45°<br>None on vertical   | Exposure<br>continuing  |              |
| Acryloid B-72 95, 0<br>Armeen C 5, 0                   | 0.1          | υ  | 2  | 10%                  | Occasional pits (large)  | Poor adhesion;<br>film peels freely   |              |
| Acrylois B-72 90, C<br>Armeen C 10, C                  | 0.1          | U  | 7  | 5%                   | Occasional pits (large)  | Poor adhesion;<br>film peels freely   |              |
| Acryloid B-72 90. 0<br>Nonyl phenoxy acetic acid 10. 0 | 0.3          | U  | 7  | 1%                   | Rare pitting (small)   | Poor adhesion;<br>film peels freely   |              |
| Acryloid B-72 90, 0<br>Glycomal MA 10, 0               | 0.2          | υ  | 7  | 11%                  | Rare pitting (small)   | V. poor adhesion:<br>film peels freely  |              |
| realized in School Strand Strand Strand                | ate ate      | 90.0<br>10.0<br>20.0<br>20.0<br>20.0<br>4<br>99.6<br>5.0<br>90.0<br>10.0 | 90.0 <0.1<br>10.0 <0.1<br>20.0 <0.1<br>20.0 <0.1<br>99.6 0.3<br>99.6 0.1<br>90.0 0.1<br>90.0 0.1<br>10.0 0.3<br>90.0 0.3 | ate 90.0 <0.1 C 20.0 | ate 90.0 <0.1 C 7 10.0 c 7 10.0 c 7 20.0 c 7 20.0 c 7 20.0 c 7 20.0 c 6 0.4 c 7 5.0 c 1 c 7 5.0 c 1 c 7 30 acetic acid 10.0 0.3 C 7 10.0 c 6 10.0 c 7 20.0 c | 90. 0 < 0. 1 C 7 90% Generally (varying, 20.0   0.4   C 7 90% Generally 20.0   0.4   C 7 90% Generally (varying, not on a 99. 6 0.3   C 2 15% Rust streate 0.4   C 7 10% Occasiona 5.0   0.1   C 7 10% Occasiona 90.0   0.1   C 7 5% Occasiona 10.0   0.3   C 7 1% Rare pitti acetic acid 10.0   0.2   C 7 1% Rare pitti | 90. 0 < 0. 1 |

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| <del>1</del>                  |  |   | <del></del>                             | I                            | ···                     | <del></del>  | ··· T                                  | 1                            | T  |  |
|-------------------------------|--|---|---|------------------------------|-------------------------|--|--|------------------------------|--|--|
| Remarks                       | Poor adhesion;<br>film peels                   | Film whitened                           | Film whitened                           | !                            | 1                       | Exposure<br>continuing   | i                                      | Film whitened                | Film whitened                              | Poor adhesion;<br>film peels freely                                      |
| % surface rusted Type of Rust | 2% Scattered pitting (small)                   | 10% Scattered pitting (small; numerous) | 10% Scattered pitting (small; numerous) | 2% Scattered pitting (small) | 1% Rare pitting (small) | Raze pitting (very small)<br>Horizontal and 45<br>None on vertical | 1% Rare pitting (small)                | 4% Scattered pitting (small) | 1% Rarc pitting (small)                    | 1% Rare pitting (small)  |
| l est<br>length<br>(days)     | 2  | 2                                       | 2                                       | 2                            | 2                       | 30   | ĩ                                      | 7                            | 7  | 2  |
| Envi-<br>ron-<br>ment         | ၁  | υ                                       | C                                       | ပ                            | ပ                       | S  | C                                      | C                            | ပ  | б  |
| Thick-<br>ness<br>(mils)      | 0.1+   | 0.2                                     | 0.2                                     | 0.3                          | 0.2                     |  | 0, 1                                   | 0.3                          | 0. 1+                                      | 0.1  |
|                               | 92. 2<br>7. 2<br>0. 6                          | 99.6                                    | 99.6                                    | 95.0<br>5.0                  | 90.0<br>10.0            | 90. 0<br>10. 0   | 90. 0<br>10. 0                         | 90. 0<br>10. 0               | 92.2<br>7.2<br>0.6                         | 95. 0  |
| Film                          | Acryloid B-72<br>Armeen C<br>Zinc linoresinate | Epon 1007<br>Zinc linoresinate          | Epon 1007<br>Zinc linoresinate          | Epon 1007<br>Armeen C        | Epon 1007<br>Armeen C   | Epon 1007<br>Armeen C  | Epon 1007<br>Nonyl phenoxy acetic acid | Epon 1007<br>Glycomul MA     | Epon 1007<br>Armeen C<br>Zinc linoresinate | Cellulose acetate butyrate<br>(Half-second)<br>Nonyl phenoxy acetic acid |
| Code                          | 65   | 35                                      | 35                                      | 48                           | 49                      | 49   | 09                                     | 61                           | 99   | 52   |

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|          |  |                | Thick-        | Envi-<br>ron- | Test<br>length | Conditi<br>% surface |   | , , , , , , , , , , , , , , , , , , ,                 |
|----------|--|----------------|---------------|---------------|----------------|----------------------|---|---|
| Code     | Film   |                | (mils)        | ment          | (days)         | rusted               | ed 1 ype of Kust  | Kemarks   |
| 53       | Cellulose acetate butyrate<br>(Half-second)<br>Nonyl phenoxy acetic acid | 90.0           | 0.2           | v             | 7              | 2%                   | Scattered pitting (small)   | Poor adhesion;<br>film peels freely                   |
| 53       | Cellulose acetate butyrate<br>(Half-second)<br>Nonyl phenoxy acetic acid | 90. 0          | 0,3+          | C             | 7              | 2%                   | Scattered pitting (small)   | Poor adhesion;<br>film peels freely                   |
| 53       | Cellulose acetate butyrate<br>(Half-second)<br>Nonyl phenoxy acetic acid | 90.0           |               | S             | 30             |                      | Rare pitting (very small)<br>Horizontal<br>None on vertical or 45   | Exposure<br>continuing                                |
| 49       | Cellulose acetate b utyrate<br>(Half-second)<br>Glycomul MA              | 90. 0          | 0.3           | ບ             | 7              | 5%                   | Underfilm rust creepage   | Film whitened:<br>Poor adhesion;<br>film peels freely |
| 29<br>7- | Cellulose acetate butyrate<br>(20-second) EAB381-20<br>Glycomul MA       | 90. 0<br>10. 0 | ~0.1<br>0.2   | ပ             | 2              | 50%<br>15%           | Generally pitted (Large) Pitting (vary'g sm. to lg.)                | V. poor adhesion;<br>film peels freely                |
| 54       | Ucar R-101   |                | 40.1          | ၁             | L              | 1%                   | Rare pitting (very small)   | Film baked at<br>115°C., 30 min.                      |
| 54       | Ucar R-101   |                | <b>≥</b> 0. 1 | ၁             | 2              | 1%                   | Rare pitting (very small)   | Film baked at<br>115°C., 30 min.                      |
| 54       | Ucar R-101   |                | <b>∠0.</b> 1  | ပ             | 30             |                      | Rare pitting (very small)<br>Vertical and 45°<br>None on horizontal | Exposure<br>continuing                                |
| 55       | Ucar R-104   |                | <b>20.</b> 1  | ນ             | 2              | 3%                   | Scattered pitting (small) and underfilm creeping rust               | Film baked at<br>115°C., 30 min.                      |
| 55       | Ucar R-104   |                | <b>20.</b> 1  | တ             | 30             | 20%                  | Generally pitted<br>(varying small to large)                        | Film baked at<br>115°C., 30 min.                      |
|          |  |                |               |               |                |                      |   |   |

|      |                      | Thick   Envi-   Test | Envi- | Г      | Condition of Panel  |                               |
|------|----------------------|----------------------|-------|--------|---|-------------------------------|
|      |                      | ness                 | ron-  | length | ron- length % surface   |                               |
| Code | Film                 | (mils)               | ment  | (days) | rusted Type of Rust   | Remarks                       |
| 99   | SR-53 Silicone resin | <0.1 C               | Ú     | 7      | 5% Scattered pitting (small; numerous)                              |                               |
| 56   | SR-53 Silicone resin | <b>40.</b> 1         | Ú     | 7      | 3% Scattered pitting (small; numerous)                              | Film baked at 115°C., 30 min. |
| 56   | SR-53 Silicone resin | <0.1                 | S     | 30     | Rare pitting (very small)<br>Horizontal and 45°<br>None on vertical | Exposure<br>continuing        |

NOTES:

C = Controlled cyclic condensation humidity cabinet.

S = Louvered shed on roof, midtown New York; Exposure started April 1963; Uncoated panels.

Rust, I day, at moderate temperatures, low relative humidity (50°F., 65°F.; 30% R.H., 70% R.H.)

Test Panel series mounted in vertical, horizontal, and 45° holders.

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